1. **Project Name:** **Inverse Process Analysis for the Acquisition of Thermophysical Data**

2. **Lead Organization:** University of Tennessee
Mechanical and Aerospace Engineering and Engineering Science, Knoxville, TN 37996-2210

3. **Principal Investigator:** Dr. Jay I. Frankel
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4. **Project Partners:** Oak Ridge National Laboratory (Adrian Sabau and Wallace Porter). Experimental facilities, computational resources, and implementation platform.
Companies: Howmet, Inc., Ford Motor, Co., Flow Science, Inc., ProCAST, Inc., NETZSCH Instruments, Inc.

5. **Date Project Initiated and FY of Effort:** 10/01/2001, FY 200

6. **Expected Completion Date**: 10/01/2004

7. **Project Technical Milestones and Schedule:**

The project objective is to increase the accuracy of thermophysical property data by additional post-processing of the experimental data.

The lack of accurate experimental data on material thermophysical properties is one of the main barriers in the analysis and design of materials processing and industrial applications. For example, in the aluminum, steel, and metalcasting industries prediction of defect formation such as shrinkage voids, microporosity, and macrosegregation is limited by inaccurate data available on fraction solid and density evolution during solidification.

The post-processing of data will consist of inverse-type computational analyses of the measurement process. By performing computational analyses, the time-temperature lags, which are inherent to the measurement process, can be estimated and their effect can be taken into account in determining more accurate thermophysical properties. The two instruments considered heat-flux differential scanning calorimeters (DSCs) and push-rod dilatometers (DILs) provide data on the amount of solid fraction and density evolution during phase changes, respectively.

Milestones are at the end of tasks.

Task ID	*Task / Milestone Description*	*Planned Completion*	*Actual Completion*	*Comments*
1	**DSC - Develop Analytical Models**			
1.2	Identify temperature dependent model parameters	6/30/02	6/30/02	completed
1.3	Develop and implement "parameter estimation" type inverse models	12/31/03		On schedule
1.4	Validate the inverse algorithm	12/31/03		On schedule
2	**Dilatometer - Develop Analytical Models**			
2.2	Develop inverse model for container and sample	6/30/02	6/30/02	completed
2.3	Implement "function estimation" type inverse methodology	12/31/03		On schedule

Task ID	*Task / Milestone Description*	*Planned Completion*	*Actual Completion*	*Comments*
2.4	Validate the inverse algorithm	12/31/03		On schedule
3	**DSC and Dilatometry Measurements**			
3.1	Additional thermocouple instrumentation of dilatometer	12/31/02	5/31/03	Completed*
3.2	Calibrate instruments	12/31/02		Ongoing
3.3	Perform DSC measurements at different cooling/heating rates	6/30/03		On schedule
3.4	Perform dilatometry measurements at different heating/cooling rates	6/30/03		On schedule
4	**Experimental Validation of proposed methodologies**			

*date changed due scheduled use of apparatus

8. **Past Project Milestones and Accomplishments:**

(1) DSC - A heat transfer model comprising the key components of the DSC system has been developed using time constants to account for thermal lags in the system. The governing equations for the model were recast as a parameter estimation problem, which was solved using an iterative algorithm based on the Function Decomposition Method (FDM). The major achievements for the DSC tasks include finding the minimum number of model parameters that describe system.

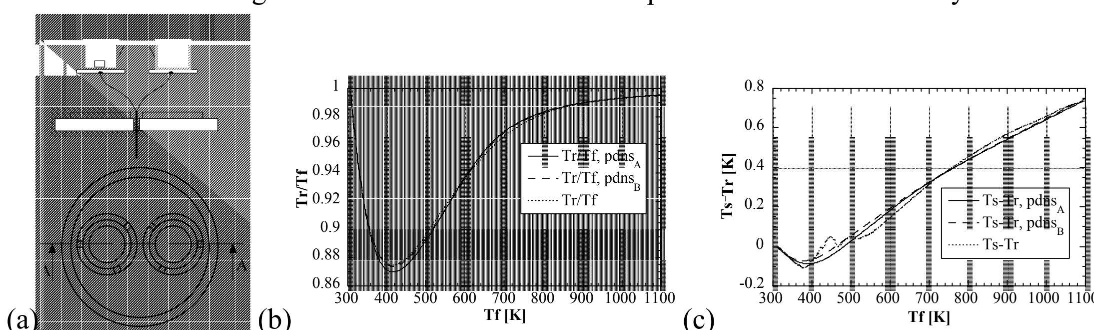

Figure 1. Schematic of the DSC system. The reference temperature (b) and the sample temperature (c) are both well predicted when the alumina disk and system asymmetry are considered in the model. *Dotted line represents the experimental data.*

(2) Dilatometer - Original dilatometer configuration requires a quasi-equilibrium data acquisition approach (i.e., slow heating rates, long experiment times). The new sample holder was designed such that can hold thermocouples and insure a uniform heat flux distribution with minimal modifications made to the rest of the dilatometer. Using the temperature data in the holder, an inverse heat conduction analysis can be performed to obtain very accurate estimates of the actual sample temperature.

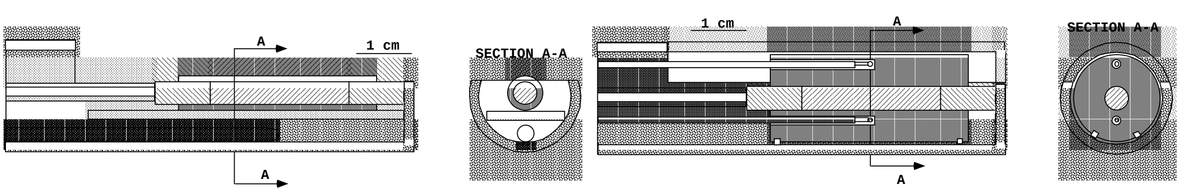

Figure 2. Schematic of original and new sample holder.

The major achievements for the Dilatometer case include the completion of (a) design and manufacture of sample holder, (b) inverse heat conduction for dilatometer configuration, and (c) development of a method for estimating thermocouple measurement error. It was estimated that the new approach to data acquisition will reduce the experiment duration by at least ten times.

The local errors were determined from typical thermocouple data based on a new method developed [3]. The error magnitude is consistent with that provided by the manufacturer. By using this error estimate, the differentiation of data can now be performed in a robust manner. The temperature rate for the two thermocouples is required in the inverse analysis of the dilatometer. The finite difference results (using raw data) shown in Fig 6 are presented for illustrative purposes only.

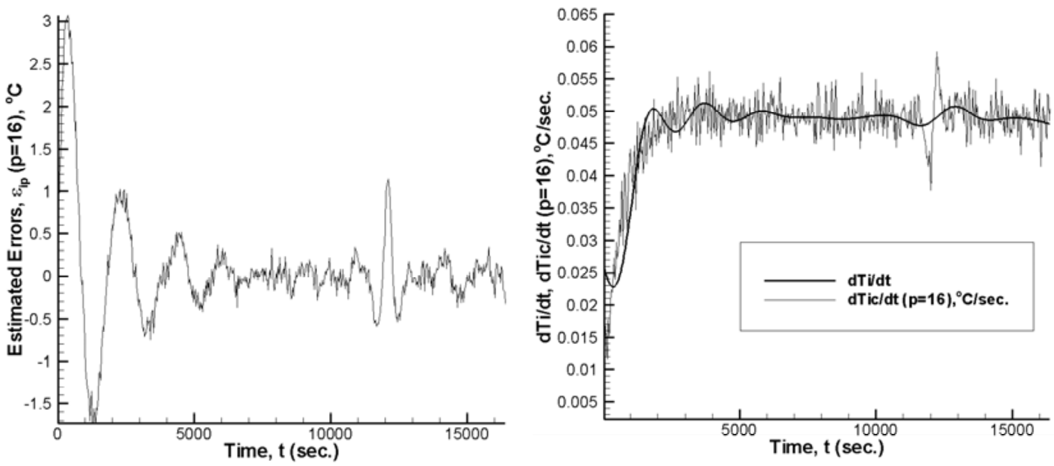

Figure 6: (a) Estimated local temperature errors using Frankel-Keyhani-Taira [3] method and (b) Estimated thermocouple heating rate using corrected data and original (raw) data based on central difference approximation.

9. **Planned Future Milestones:** All future tasks are on schedule.

Task ID	*Task / Milestone Description*	*Planned Completion*
1	**DSC - Develop Analytical Models**	
1.3	Develop and implement "parameter estimation" type inverse models	12/31/03
1.4	Validate the inverse analysis algorithms	
2	**Dilatometer - Develop Analytical Models**	
2.3	Develop inverse model for container and sample	12/31/03
2.4	Validate the inverse analysis algorithms	12/31/03
3	**DSC and Dilatometry Measurements**	
3.3 3.4	Perform DSC and dilatometry measurements at different cooling/heating rates	6/30/03
4	**Experimental Validation of proposed methodologies**	
4.1	Determine new property data by performing inverse analysis	3/31/04
4.2	Assess the accuracy of using the new property data by comparing experimental data with numerical simulation results for cooling curves and shrinkage predictions	6/30/04
5	**Write report on experimental and computational procedures**	
5.1	Write flow chart of computational methodologies developed	8/31/04
5.2	Write experimental procedures for DSC and dilatometer operation	9/31/04
5.3	Complete Final Report	9/31/04

10. **Issues/Barriers:** The major issues encountered were modeling the heat exchange between different parts for the DSC assembly, evolution of furnace temperature, and accounting for the systematic error introduced in the DSC instrument due to its asymmetry. The asymmetry in the system is now considered through an analytical formulation. A major problem is to determine the rest of the model parameters through the inverse analysis. The major deviation from original scope was the need to develop a new method for predicting local errors in measured data [3]. This was required for the inverse method since it allows for the determination of the optimal prediction from a family of solutions.

11. **Intended Market and Commercialization Plans/Progress:**

All computational and experimental procedures that will be developed to improve the acquisition of thermophysical properties considered will be published and presented to conferences organized by professional associations. The companies participating in this work will have access to the new thermophysical property data generated in this program. ProCAST is distributing with its software a material property database, disseminating the information across industries.

The hardware and software developed for the dilatometer will be made available to the DOE user lab facilities, such as the High Temperature Materials Laboratory (HTML) of ORNL.

The results of this program will be shared with instrument manufacturers. The instrument manufacturers will implement their appropriate post-processing analysis procedures and/or design changes in their instruments based on the methodologies outlined in our program.

12. **Patents, publications, presentations**

[1] Osborne, G.E., J.I. Frankel, and A.S. Sabau, “A New Parameter Estimation Method for DSC Thermodynamic Property Evaluation, Part I: Analytic Developments”, *22^th IASTED International Conference on Modeling, Identification and Control* (MIC 2003), Innsbruck, Austria, February 10-13, 2003.

[2] Osborne, G.E., J.I. Frankel, and A.S. Sabau, “A New Parameter Estimation Method for DSC Thermodynamic Property Evaluation, Part II: Runge-Kutta Implementation and Numerical Results”, *22^th IASTED International Conference on Modeling, Identification and Control* (MIC 2003), Innsbruck, Austria, February 10-13, 2003.

[3] Frankel, J.I., M. Keyhani, K. Taira, ”In-Phase Error Estimation of Experimental Data and Optimal First Derivatives“, *41^th AIAA Aerospace Sciences Meeting and Exhibit*, Reno, NV, January 14-17, 2003 (AIAA-2003-0516).

13. Highlight:

Inverse Process Analysis for the Acquisition of Thermophysical Property Data

Frankel, J.I., Keyhani, M., Baker, A.J. - University of Tennessee, Knoxville

Sabau, A.S, and Porter, W.D. - Oak Ridge National Laboratory

Objective: Increase the accuracy of thermophysical property data by additional post-processing of the experimental data

Background: The lack of accurate experimental data on material thermophysical properties is one of the main barriers in the analysis and design of materials processing and industrial applications. For example, in the aluminum, steel, and metalcasting industries prediction of defect formation such as shrinkage voids, microporosity, and macrosegregation is limited by inaccurate data available on fraction solid and density evolution during solidification.

Accomplishments:

(1) DSC- A heat transfer model has been developed to account for thermal lags in the DSC system. The model parameters are time constants associated with appropriate heat transfer mechanism among the key system components.

(2) Dilatometer - The new sample holder was designed such that can hold thermocouples and insure a uniform heat flux distribution with minimal modifications made to the rest of the dilatometer.

Figure 1. Cell mounting for typical heat-flux type DSC system used for high-temperature applications.

(a)

(b)

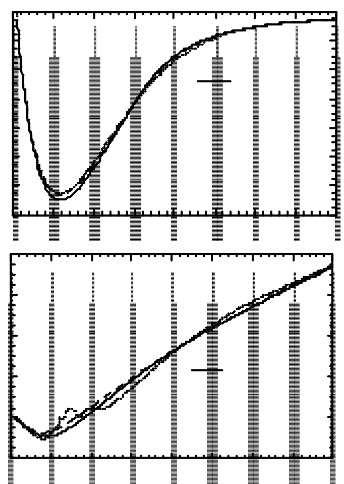

Figure 2. The reference temperature (a) and the sample temperature (b) are both well predicted when the alumina disk and system asymmetry are considered in the model.

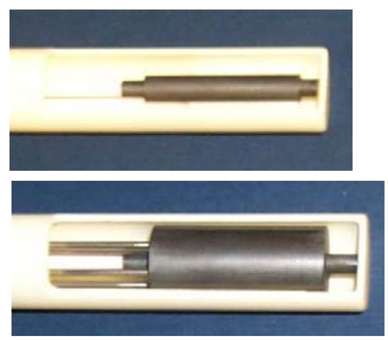

Figure 3. Old and new sample containers for dilatometer. The container is enlarged to provide appropriate conduction path. Four thermocouples are embedded into the sample container.

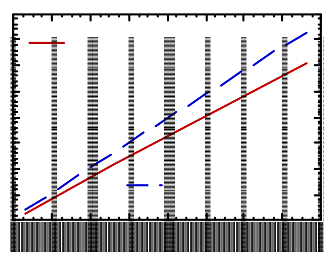

Figure 4. At high heating rates, dilatometer sample is almost isothermal while large temperature differences are expected between the container and sample. *The inverse analysis will account for these large temperature differences, decreasing the experiment time by a factor of ten.*

Industrial support

ProCAST, Inc.
Flow Science, Inc.
Howmet Research Corp.
Ford Motor Co.
Netzsch Instruments, Inc.

Future Tasks

¥Validate the inverse analysis algorithms.

¥Assess the accuracy of using the new property data by comparing experimental data with numerical simulation results for cooling curves and shrinkage during casting.

Acknowledgements

Energy Efficiency and Renewable Energy, U.S. Department of Energy.